Patent claims:

1. A process for the

a) isolation of the 3R,2'R stereoisomer of glycopyrronium bromide or iodide (formula II: A = Br or I),

from the diastereomer mixture consisting of the 3R,2'R isomer and 3R,2'S isomer (formula III)

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or from the diastereomer mixture consisting of the 3R,2'R isomer and 3S,2'R isomer (formula IIIb)

or

b) isolation of the 3S,2'S isomer (formula IV: A = Br or I),

from the diastereomer mixture consisting of the 3S,2'R isomer and 3S,2'S isomer (formula V)

or from the diastereomer mixture consisting of the 3R,2'S isomer and 3S,2'S isomer (formula Vb)

or

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c) isolation of the 3R,2'S stereoisomer of the thienyl analog of glycopyrronium (formula $VI: A = Br \ or \ I$),

from the diastereomer mixture consisting of the 3R,2'S isomer and 3R,2'R isomer (formula VII)

or from the diastereomer mixture consisting of the 3R,2'S isomer and 3S,2'S isomer (formula VIIb)

or

d) isolation of the 3S,2'R isomer (formula VIII: A = Br
or I),

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from the diastereomer mixture consisting of the 3S,2'S isomer and 3S,2'R isomer (formula IX)

or from the diastereomer mixture consisting of the 3S,2'R isomer and 3R,2'R isomer (formula IXb)

characterized

- in that in the quaternization to give the abovementioned diastereomer mixtures a suitable solvent is used in order to obtain the stereoisomer to be isolated in each case in enriched form as a precipitate and/or
- in that the diastereomer mixtures of the quaternary compounds described above are recrystallized in a suitable solvent or solvent mixture and in this process the isomer desired in each case is obtained in enriched form.
- 15 2. The process as claimed in claim 1, in which solvent having a water content which leads to only the desired diastereomer being obtained in crystalline form is used, while the other diastereomer remains in solution or is obtained as an oil.
- 20 3. The process as claimed in claim 1 or 2, in which in the quaternization to give said diastereomer mixtures of the quaternary salts a suitable solvent, such as, for example, isopropanol or acetone, is used and thus said stereoisomers are isolated in enriched form in the resulting precipitate.
 - 4. The process as claimed in claim 1, 2 or 3, in which a solvent in which the diastereomer mixture dissolves readily is used for the re-crystallization and a second

- solvent causing crystallization is added in order to bring about crystallization.
- 5. The process as claimed in claim 4, in which methanol and/or ethanol are/is preferably used for dissolving and crystallization is brought about using ethyl acetate and/or tert-butyl methyl ether.

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- 6. The process as claimed in claim 1, 2 or 3, in which for the recrystallization the diastereomer mixture is dissolved in a heated solvent and crystallization takes place by cooling.
- 7. The process as claimed in claim 6, in which the diastereomer mixture is dissolved at boiling heat in 2-propanol or ethanol and crystallization takes place on cooling to room temperature or below.
- 15 8. The process as claimed in claims 1-7 preferably for the enrichment of the 3R,2'R isomer of glycopyrronium bromide.
 - The process as claimed in one of the preceding claims as 9. prepurification stage for obtaining а primary enrichment of diastereomers or, if enrichment has already taken place, to give a further increase in diastereomer purity.
- 10. The process as claimed in one of the preceding claims, solvents preferably selected from the group consisting of branched and unbranched alcohols having a low molecular weight, such as methanol, ethanol, isopropanol, 1-propanol, tert-butanol, isobutanol, n-butanol, and also acetone, butanone or acetonitrile, in each case pure or as mixtures with other solvents, being used.
- 30 11. The process as claimed in one of the preceding claims, a solvent having a water content of preferably less than approximately 5%, even more preferably approximately 0.5-2%, most preferably approximately 1%, being used in the quaternization.

12. The process as claimed in one of the preceding claims, a solvent having a water content of preferably approximately 0.2-3%, more preferably approximately 0.5%, being used in the recrystallization.